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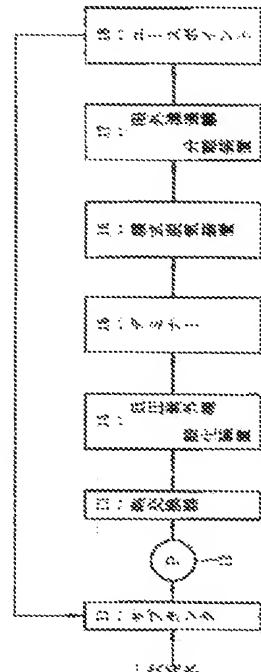
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(54)【発明の名称】 超純水製造装置

(57)【要約】

【課題】 蒸存微素量が著しく低い超純水を製造する。
【解決手段】 前処理工程より得られた一次純水をサブタンク1、ポンプ12、熱交換器13、低圧紫外線酸化装置14、イオン交換装置15、膜式脱気装置16及び紫外通過膜分離装置17に順次に通水し、得られた極低溶存微素の超純水をユースポイント18に送る。



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【特許請求の範囲】

【請求項1】 一次純水を導入し、少なくとも紫外線照射装置とイオン交換純水装置とを有するサブシステムに通水して超純水を得る超純水製造装置において、該イオン交換純水装置の後段に膜式脱気装置を配置したことを特徴とする超純水製造装置。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は超純水製造装置に係り、特に溶存酸素濃度がきわめて低い超純水を得る超純水製造装置に関するものである。

【0002】

【従来の技術】従来、半導体洗浄用水として用いられている超純水は、図2に示すように前処理システム1、一次純水システム2及びサブシステム3から構成される超純水製造装置で原水（工業用水、雨水、井戸水等）を処理することにより製造されている。図2において、各システムの役割は次の通りである。

【0003】 過濾、加圧浮上（沈殿）、逆浸透装置等よりも前処理システム1では、原水中の懸濁物質やコロイド物質の除去を行う。逆浸透装置分離装置、脱気装置及びイオン交換装置（膜床式又は4床式）を備える一次純水システム2では原水中のイオンや有機成分の除去を行う。なお、逆浸透装置分離装置では、塩類除去のほかにイオン性、コロイド性のTOCを除去する。イオン交換装置では、塩類除去のほかにイオン交換樹脂によって吸着又はイオン交換されるTOC成分を除去する。脱気装置（窒素脱気又は真空脱気）では溶存酸素の除去を行う。

【0004】 縫交換器、低圧紫外線照射装置、ボリッシャー（非再生式イオン交換樹脂装置）及び紫外線過濾分離装置を備えるサブシステム4では、水の純度をより一層高め超純水にする。なお、低圧紫外線照射装置では、低圧紫外線ランプより出される185nmの紫外線によりTOCを有機物さらにはCO₂まで分解する。分解された有機物及びCO₂は後段のイオン交換樹脂で除去される。紫外線過濾分離装置では、微小粒子が除去されイ*



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* オン交換樹脂の流出粒子も除去される。

【0005】

【発明が解決しようとする課題】超純水中の溶存酸素は、シリコンウェハーの自然酸化膜の厚さをコントロールする上で重要な因子であるが、上記従来の超純水製造装置によれば、溶存酸素濃度から~10ppb程度となってしまい、さらに低い溶存酸素値が要求されるケースでは要求水質を満足できなくなる。

【0006】 本発明は、超純水中の溶存酸素を効率的に除去できる超純水製造装置を提供することを目的とする。

【0007】

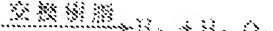
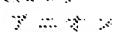
【課題を解決するための手段】本発明の超純水製造装置は、一次純水を導入し、少なくとも紫外線照射装置（紫外線照射装置）とイオン交換純水装置とを有するサブシステムに通水して超純水を得る超純水製造装置において、該イオン交換純水装置の後段に膜式脱気装置を配置したことを特徴とする。

【0008】 本発明者らは、図2に示されるような超純水製造装置における溶存酸素濃度の推移を調べた結果、サブシステム内の低圧紫外線照射装置出口で溶存酸素濃度は低下するが、後段のボリッシャー出口で再度低圧紫外線照射装置入口の溶存酸素濃度まで（場合によつてはそれ以上に）上昇してしまい、結果的に低圧紫外線照射装置の溶存酸素除去効果が表れないことを確認した。即ち、一次純水システムの流出水の溶存酸素濃度は約5~10ppbであり、この水が低圧紫外線照射装置で処理されることにより、溶存酸素濃度は0~5ppbに低減するが、ボリッシャー出口水の溶存酸素濃度は再び5~10ppb程度に上昇しており、結果として得られる超純水の溶存酸素濃度は5~10ppbとなる。

【0009】 本発明者らは、この現象について観察検討した結果、下記(1)式のような反応が低圧紫外線照射装置からボリッシャーの間で生じていることを見出した。

【0010】

(化1)



する。しかし、生成したR.O.（ボリッシャー（特化ボリッシャー内のアセチル交換樹脂）の接触触媒作用で分解され、再度O₂が発生するため、ボリッシャー出口水の溶存酸素濃度が上昇する。

【0012】 本発明は、紫外線照射装置からイオン交換装置を経ることにより発生した溶存酸素を膜式脱気装置で除去することにより、得られる超純水中的溶存酸素値を低下させるようにしたものである。

【0013】

【発明の実施の形態】以下、図面を参照して本発明を詳

補に説明する。

【0014】図1は本発明の超純水製造装置の一実施例を示す系統図である。

【0015】各種前処理工程より得られた一次純水(通常の場合、TOC濃度2ppb以下の純水)をサブタンク11、ポンプ12、熱交換器13、低圧紫外線酸化装置14、イオン交換装置(本実施例ではアノン交換樹脂とカチオン交換樹脂とをイオン負荷に応じて混合したデミナー)15、膜式脱気装置16及び限外透達膜分離装置17に順次に通水し、得られた極低溶存酸素の超純水をユースポイント18に送る。

【0016】膜式脱気装置としては、脱気膜の一方の側に水を流し、他方の側を真空ポンプで排気し、溶存酸素を膜を透過させて真空側に移行させて除去するようにしたもののが用いられる。なお、この膜の真空側には若干の水分が脱気膜を透過して出てくるので、この真空側に窒素等のガスを流し、水分を除去して膜性能の低下を防止するのが好ましい。N₂流量は一定でも良く、密閉させても良い。

【0017】脱気膜は、酸素、窒素、蒸気等のガスは通過するが水は通過しない膜であれば良く、例えば、シリコンゴム系、ポリテトラフルオロエチレン系、ポリオレフィン系、ポリウレタン系等がある。この脱気膜としては市販の各種のものを用いることができる。

【0018】この膜式脱気装置の真空度は5~70Torr、N₂等のガスの真空側流量は水流量の1~25%とするのが好ましい。なお、この運転条件は、膜性能により任意に設定できるが、通常上記のような範囲が好ましい。真空度が過度に低いと脱気効率が低下し、逆に過度に大きいと膜を通して水も透過側に出てきて効率が悪くなる。N₂流量は過度に少ないと水分除去が十分でなく脱気効率が低下し、大きすぎると真空度が上がり脱気効率が低下する。

【0019】なお、図1の構成は本発明の一例であり、本発明はサブシステムにおいて紫外線酸化装置、イオン交換装置及び膜式脱気装置を備えている限り各種の機器を組み合わせることができる。例えば、膜脱気の後に限外透達(UF)装置や逆浸透膜装置を設置しても良い。また、原水をpH4.5以下の酸性下でかつ、酸化剤存在下で加熱分解処理して原水中の尿素及び他のTOC成分を分解した後、脱イオン処理する装置を組み込むこともできる。紫外線酸化装置、イオン交換装置、膜式脱気装置は多段に設置されても良い。なお、図1のよう、膜式脱気装置の後段にUF装置を設置することにより、脱気膜で発生した微粒子を除去し、ユースポイントへの持ち込みを阻止できる。

【0020】本発明においては、紫外線酸化装置で紫外

線照射された水をイオン交換樹脂に接触させた後、脱気処理することが重要であり、膜式脱気装置を紫外線酸化装置とイオン交換樹脂との間に配置しても、得られる超純水中の溶存酸素は低減しない。

【0021】

【実施例】以下に比較例及び実施例を挙げて本発明をより具体的に説明する。

【0022】比較例1

原水(厚木市水: TOC 700~800 ppb、溶存酸素 8000 ppb、電導度 240 μS/cm)を図2の装置によって処理して超純水を製造した。

【0023】実施例1~3

サブシステムを、膜式脱気装置を組み込んだ図1のものとし、膜式脱気装置の運転条件及び通水量を表1の通りとしたほかは比較例1と同様にして超純水を製造した。

【0024】この比較例及び実施例における各装置の仕様は下記の通りである。

【0025】低圧紫外線照射酸化装置: 80W×4本(0.32 kW)

(発生波長: 185 nm及び254 nm)

ボリッシャー(デミナー): カチオン交換樹脂とアノン交換樹脂とを混合充填した混床式イオン交換装置 SV=7.0~8.0 hr⁻¹

限外透達膜分離装置: KU-1010(栗田工業株式会社製)

脱気膜: Hoechst Celanese 製Liqui-Cel-1 1本

ハウジング: 5PC-H-120

カートリッジ: 5PC-H-118, SN: 1221

【0026】

【表1】

	水流量 (m ³ /hr)	膜式脱気装置	
		真空度(Torr)	N ₂ 濃度(mmol/l)
実施例1	1.8	70	0.5
実施例2	4	55	0.5
実施例3	3	65	0.5
比較例1	3	—	—

【0027】得られた超純水の電導度と低圧紫外線酸化装置以降の各装置の流出水の溶存酸素は表2に示す通りであった。

【0028】

【表2】

	溶存酸素濃度 (ppb)					超純水 製造率 (μS/cm)
	一次純水	紫外線強化 装置出口	バケ出ロ	脱気装置 出ロ	UF出ロ	
実施例1	<5	<5	9	1	1	18.24
実施例2	<5	<5	7	<1	<1	18.24
実施例3	<5	<5	7	<1	<1	18.24
比較例1	<5	<5	7~10	—	7~11	18.24

【0029】以上より、本発明例による溶存酸素濃度が極めて低い超純水が得られることが明らかである。

【0030】

【発明の効果】以上詳述した通り、本発明の超純水製造装置によると、溶存酸素濃度が著しく低い超純水を製造できる。

【図面の簡単な説明】

【図1】本発明の超純水製造装置の一実施例を示す系統図である。

【図2】従来の超純水製造装置を示す系統図である。

【符号の説明】

* 1 前処理システム

2 一次純水システム

3 サブシステム

1.1 サブタンク

1.2 ポンプ

1.3 熱交換器

1.4 高圧紫外線強化装置

1.5 イオン交換装置（デミナ...）

2.9 1.6 脱気装置

1.7 紫外線強化分離装置

*

【図1】

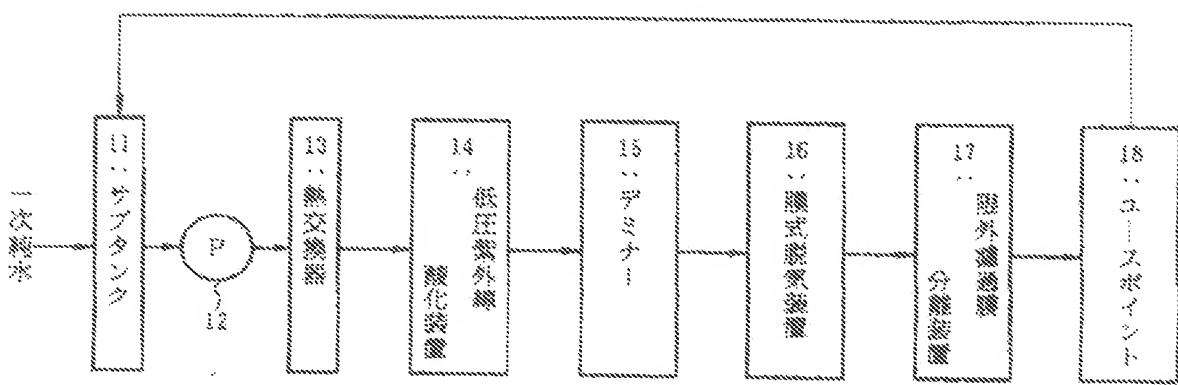
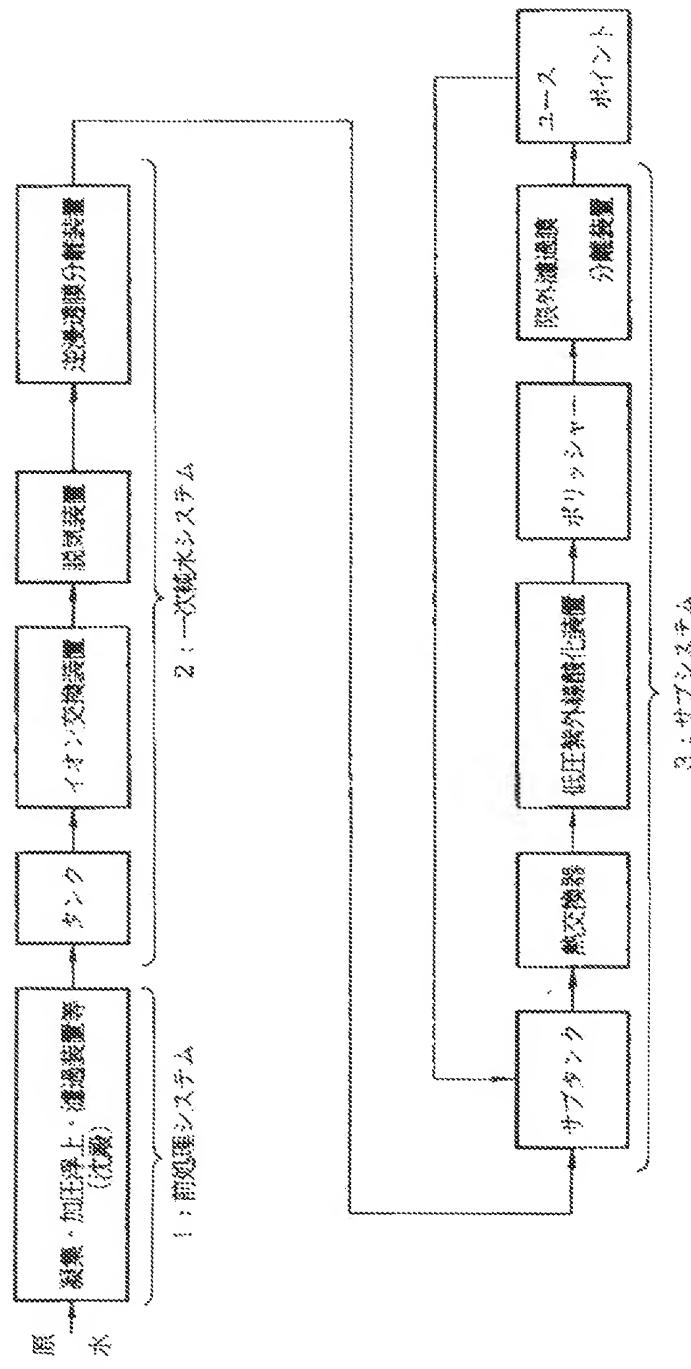


図2



PATENT ABSTRACTS OF JAPAN

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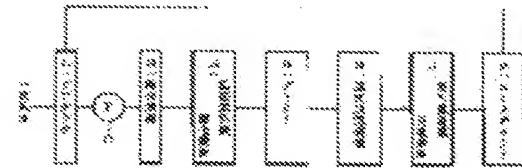
(72)Inventor : FURUKAWA MASAHIRO
KOIZUMI MOTOMU

(54) ULTRAPURE WATER PREPARING APPARATUS

(57)Abstract:

PROBLEM TO BE SOLVED: To efficiently eliminate dissolved oxygen in ultrapure water by arranging a membrane type deaeration device at the rear stage of an ion exchange pure water device in an apparatus wherein the primary pure water is introduced and the water is fed into a subsystem at least with a UV ray irradiation oxidation device and the ion exchange pure water device to obtain an ultrapure water.

SOLUTION: In an ultrapure water preparing apparatus for preparing ultrapure water used for cleaning water for semiconductor, the primary pure water obtd. from various pretreatment processes is successively passed through a subtank 11, a pump 12, a heat exchanger 13, a low pressure UV oxidation device 14, an ion exchange device 15, a membrane type deaeration device 16 and an ultrafiltration membrane separation device 17 and the obtd. ultrapure water with extremely low dissolved oxygen is sent to a use point 18. In the membrane type deaeration device 16, water is made to flow on one side of the membrane and another side is evacuated by means of a vacuum pump to permeate dissolved oxygen through the membrane and to transfer the oxygen on the vacuum side for elimination. It is pref. that the degree of vacuum of this deaeration device 16 is regulated to 55–70Torr and the flow rate of gas such as N₂ on the vacuum side is regulated to 5–25% of the amt. of water.



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CLAIMS

[Claim(s)]

[Claim 1] Ultrapure water equipments characterized by having arranged the film type deaerator in the latter part of this ion-exchange demineralizer in the ultrapure water equipments which introduce primary pure water, let water flow to the subsystem which has a UV irradiation oxidation system and an ion-exchange demineralizer at least, and obtain ultrapure water.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the ultrapure water equipments with which ultrapure water equipments are started, especially dissolved oxygen concentration obtains very low ultrapure water.

[0002]

[Description of the Prior Art] Conventionally, the ultrapure water used as semi-conductor rinse water is manufactured by processing raw water (industrial water, a city water, well water, etc.) with the ultrapure water equipments which consist of a pretreatment system 1, a primary-pure-water system 2, and a subsystem 3 as shown in drawing 2 . In drawing 2 , the role of each system is as follows.

[0003] In the pretreatment system 1 which consists of condensation, floatation (precipitate), a filter, etc., clearance of the suspended solid in raw water or a colloid substance is performed. In the primary-pure-water system 2 equipped with a reverse osmotic membrane decalibrator, a deaerator, and an ion exchange unit (4 floor 5 column a mixed bed type or type), the ion in raw water and clearance of an organic component are performed. In addition, in a reverse osmotic membrane decalibrator, TOC of ionicity and colloid nature other than salts clearance is removed. In an ion exchange unit, the ion exchange resin other than salts clearance removes adsorption or the TOC component by which the ion exchange is carried out. Dissolved oxygen is removed in a deaerator (nitrogen deaeration or vacuum desiring).

[0004] In the subsystem 4 equipped with a heat exchanger, low voltage ultraviolet ray oxidation equipment, a polisher (non-reproducing formula ion-exchange resin equipment), and an ultrafiltration membrane decalibrator, the purity of water is raised further and it is made ultrapure water. in addition, the 185nm ultraviolet rays taken out with low voltage ultraviolet ray oxidation equipment from a low voltage ultraviolet ray lamp --- TOC --- an organic-acid pan --- CO₂ up to --- it decomposes. The organic substance and CO₂ which were decomposed It is removed by latter ion exchange resin. In an ultrafiltration membrane decalibrator, a minute particle is removed and the runoff particle of ion exchange resin is also removed.

[0005]

[Problem(s) to be Solved by the Invention] Although the dissolved oxygen in ultrapure water is a factor important when controlling the thickness of the natural oxidation film of a silicon wafer, dissolved oxygen concentration turns into 5 ~ 10ppb extent, and it becomes impossible to be satisfied with the case where a still lower dissolved oxygen value is required of demand water quality according to the above-mentioned conventional ultrapure water equipments.

[0006] This invention aims at offering the ultrapure water equipments from which the dissolved oxygen in ultrapure water is efficiently removable.

[0007]

[Means for Solving the Problem] The ultrapure water equipments of this invention are characterized by having arranged the film type deaerator in the latter part of this ion-exchange demineralizer in the ultrapure water equipments which introduce primary pure water, let water flow to the subsystem which has a UV irradiation oxidation system (ultraviolet ray oxidation equipment) and ion-exchange demineralizer at least, and obtain ultrapure water.

[0008] Although dissolved oxygen concentration once fell at the low voltage ultraviolet-ray-oxidation-equipment outlet in a subsystem as a result of this invention persons' investigating transition of the dissolved oxygen concentration in ultrapure water equipments as shown in drawing 2 , it went up again at the latter polisher outlet to the dissolved oxygen concentration of a low voltage ultraviolet-ray-oxidation-equipment inlet port (more than it curves depending on the case), and checked that the dissolved oxygen clearance effectiveness of low voltage ultraviolet ray oxidation equipment did not appear as a result. That is, although the dissolved oxygen concentration of the effluent of a primary-pure-water system is about five to 10 ppb and dissolved oxygen concentration is reduced to 0 ~ 5ppb by processing this water with low voltage ultraviolet ray oxidation equipment, the dissolved oxygen concentration of polisher outlet water is going up to 5 ~ 10ppb extent again, and the dissolved oxygen concentration of the ultrapure water obtained as a result serves as 5 ~ 10ppb.

[0009] this invention persons found out that a reaction like following the (1) type had arisen from low voltage ultraviolet ray oxidation equipment between polishers, as a result of examining this phenomenon wholeheartedly.

[0010]

[Formula 1]



[0011] That is, although water (H₂O) and TOC exist at a low voltage ultraviolet-ray-oxidation-equipment inlet port, when TOC in primary pure water is very low, the amount of UV irradiation in ultraviolet ray oxidation equipment becomes superfluous from a design value. (For example, when primary pure water of TOC5ppb flows into the ultraviolet ray oxidation equipment which made the amount of UV irradiation the dose corresponding to TOC1(ppb), the amount of UV irradiation becomes superfluous by TOC5ppb.) And H₂O is set to H₂O₂ (hydrogen peroxides) through OH radical by the exposure of these superfluous ultraviolet rays, and since dissolved oxygen is used for TOD decomposition, dissolved oxygen concentration falls seemingly. However, H₂O₂ generated is decomposed by the contact catalysis of a polisher (especially anion exchange resin in a polisher), and is O₂ again. Since it generates, the dissolved oxygen concentration of polisher outlet water goes up.

[0012] It is made for this invention to reduce the dissolved oxygen value in the ultrapure water obtained by removing the dissolved oxygen generated by passing along an ion exchange unit from ultraviolet ray oxidation equipment with a film type deaerator.

[0013]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail with reference to a drawing.

[0014] Drawing 1 is the schematic diagram showing one example of the ultrapure water equipments of this invention.

[0015] It lets flow the primary pure water (in the case of usual pure water of 2 or less ppb of TOC concentration) obtained from various head end processes one by one to the subtank 11, a pump 12, a heat exchanger 13, low voltage ultraviolet ray oxidation equipment 14, an ion exchange equipment (DEMINA which mixed an anion exchange resin and cation exchange resin according to the ion load in this example) 15, the film type deaerator 16, and the ultrafiltration membrane decalculator 17, and the ultrapure water of the obtained super-low dissolved oxygen is sent to the point of use 18.

[0016] What exhaust a sink and another side side for water with a vacuum pump to one deaeration film side, and make the film penetrates, and dissolved oxygen is made to shift to a vacuum side, and was removed as a film type deaerator is used. In addition, since some moisture penetrates the deaeration film and comes out to the vacuum side of this film, it is desirable to remove a sink and moisture for gas, such as nitrogen, and to prevent lowering of membranous ability to this vacuum side. N2 Regularity is sufficient as a flow rate and it may be fluctuated.

[0017] Although gas, such as oxygen, nitrogen, and a steam, passes the deaeration film, water has a silicone rubber system, a polytetrafluoroethylene system, a polyolefine system, a polyurethane system, etc. that what is necessary is just the film which is not penetrated. Various kinds of commercial things can be used as this deaeration film.

[0018] the degree of vacuum of this film type deaerator --- 55 ~ 70Torr and N2 etc. --- as for the amount of vacuum side streams of gas, it is desirable to carry out to 5 ~ 25% of a water flow rate. In addition, although this service condition can be set as arbitration by membranous ability, its usually above range is desirable. If a degree of vacuum is too low, deaeration effectiveness will fall, if too conversely large, through the film, water will also appear in a transparency side and effectiveness will worsen. N2 If there are too few flow rates, moisture clearance will not be enough, deaeration effectiveness will fall, if too large, a degree of vacuum will not go up but deaeration effectiveness will fall.

[0019] In addition, the configuration of drawing 1 is an example of this invention, and this invention can combine various kinds of devices, as long as it has ultraviolet ray oxidation equipment, the ion exchange unit, and the film type deaerator in the subsystem. For example, ultrafiltration (UF) equipment and reverse osmotic membrane equipment may be installed after film deaeration. Moreover, after carrying out thermal decomposition processing of the raw water under with a pH of 4.5 or less acidity and oxidizer existence and decomposing the urea and other TOC components in raw water, the equipment which carries out deionization processing is also incorporable. Ultraviolet ray oxidation equipment, an ion exchange unit, and a film type deaerator may be installed in multistage. In addition, like drawing 1, by installing UF equipment in the latter part of a film type deaerator, the particle generated by the deaeration film is removed and the drag-in to the point of use can be prevented.

[0020] In this invention, after contacting the water by which UV irradiation was carried out with ultraviolet ray oxidation equipment on ion exchange resin, even if it is important to carry out film deaeration processing and it arranges a film type deaerator between ultraviolet ray oxidation equipment and ion exchange resin, the dissolved oxygen in the ultrapure water obtained is not reduced.

[0021]

[Example] The example of a comparison and an example are given to below, and this invention is more concretely explained to it.

[0022] Example of comparison 1 raw water (Atsugi city water TOC700 ~ 800ppb, dissolved oxygen 8000ppb, electric conductivity S/cm of 240micro) was processed with the equipment of drawing 2, and ultrapure water was manufactured.

[0023] Made one to example 3 subsystem into the thing of drawing 1 incorporating a film type deaerator, and the service condition and the amount of water flow of a film type deaerator were carried out as a table 1, and also ultrapure water was manufactured like the example 1 of a comparison.

[0024] The specification of each equipment in this example of a comparison and example is as follows.

[0025] Low-voltage UV-irradiation oxidation system: 80Wx4 ** (0.32kW)

(Generating wavelength: 185nm and 254nm)

polisher (DEMINA): --- mixed bed type ion exchange unit SV=70 which carried out mixed filling of cation-exchange resin and anion exchange resin ~ 80hr~1 ultrafiltration-membrane decollator: --- KU-1010 (Kurita Water Industries, Ltd. make)

deaeration film: Hoechst Celanese Make Liqui-Cel 1 housing: --- 5PCH-120 cartridge: --- 5PCH-118 and SN: --- 1221 [0026]

[A table 1]

	水流量 (m ³ /hr)	膜式脱氷新装置	
		真空度 (Torr)	N ₂ 流量 (Nm ³ /h)
実施例1	1. 8	7.0	0. 6
実施例2	4	6.5	0. 6
実施例3	3	6.5	0. 6
比較例1	3	—	—

[0027] The dissolved oxygen of the electric conductivity of the obtained ultrapure water and the effluent of each equipment after low voltage ultraviolet ray oxidation equipment was as being shown in a table 2.

[0028]

[A table 2]

	溶存酸素 (ppb)					超純水電導度 (μS/cm)
	一次純水	紫外線酸化 装置出口	分岐出口	脱氷新装置 出口	UF出口	
実施例1	<5	<5	9	1	1	18.24
実施例2	<5	<5	7	<1	<1	18.24
実施例3	<5	<5	7	<1	<1	18.24
比較例1	<5	<5	7~10	—	7~11	18.24

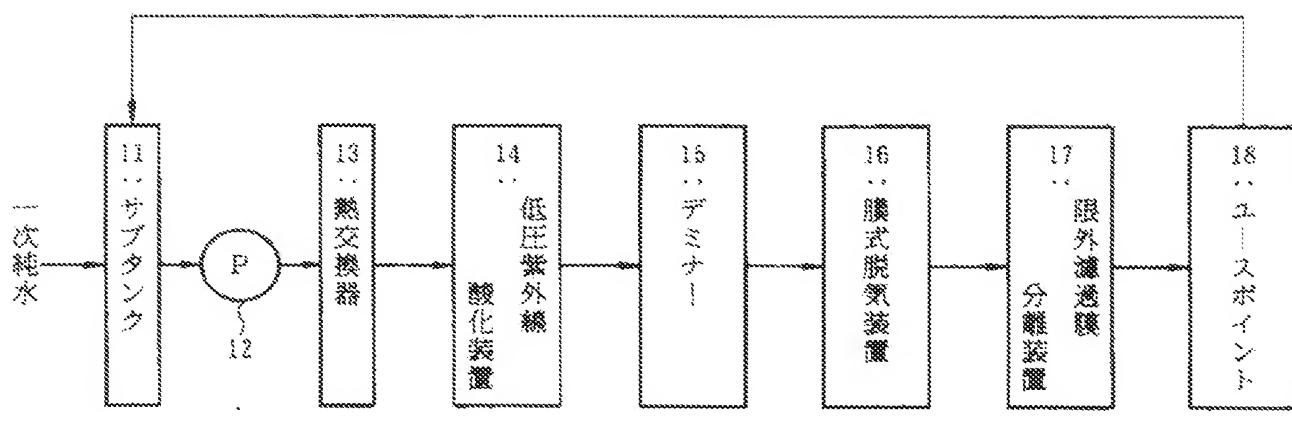
[0029] According to the example of this invention, it is clearer than a table 2 that ultrapure water with very low dissolved oxygen concentration is obtained.

[0030]

[Effect of the Invention] According to the ultrapure water equipments of this invention, dissolved oxygen concentration can manufacture remarkable low ultrapure water as explained in full detail above.

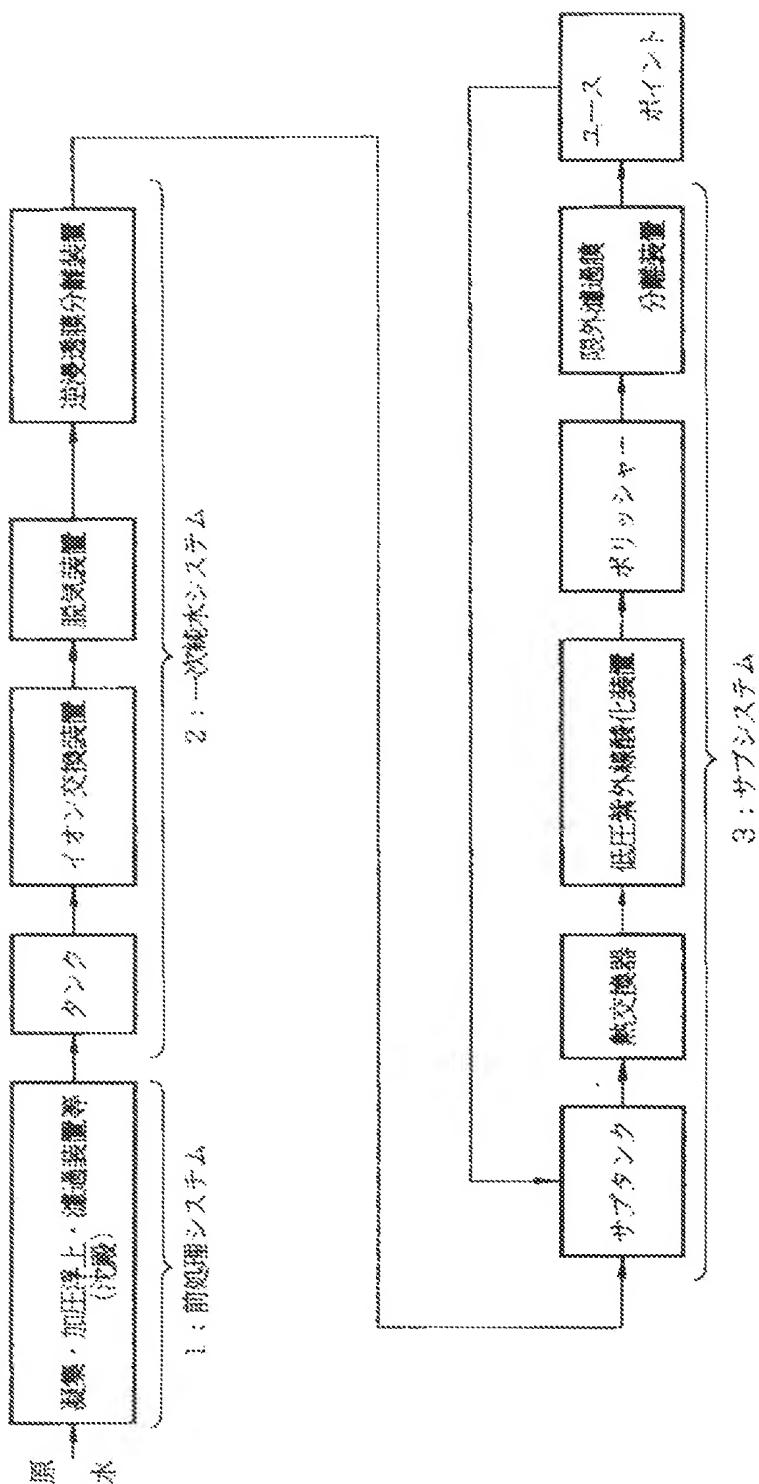
[Translation done.]

Drawing selection drawing 1



[Translation done.]

Drawing selection drawing 2



[Translation done.]